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AUTHORITY

USAEC ltr, 15 Jul 1971

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RADIO CORPORATION OF AMERICA

UNCLASSIFIED

STUDY OF WATER SOLUBLE RESIDUE FLUXES

Quarterly Report #1

Signal Corps Contract #DA-28-043-AMC-01365(E)

First Quarterly Progress Report 1 June 1965 to 31 August 1965

U.S. Army Electronics Research and Development Laboratory,

Fort Monmouth, New Jersey

August 31, 1965

Materials Standards

Central Engineering

Defense Electronic Products

UNCLASSIFIED

STUDY OF WATER SOLUBLE RESIDUE FLUXES

Quarterly Report #1

Signal Corps Contract #DA-28-043-AMC-01365(E)

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The object of this Study of Water Soluble Residue Fluxes is to investigate and evaluate various solder fluxes not presently covered by MIL-F-14256, with an end goal of accumulating comparative data upon which to base the selection criteria of a Military Procurement Specification.

Report Prepared by: O. D. Black

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1. PURPOSE

The purpose of this Study of Water Soluble Residue Fluxes is to investigate and evaluate various solder fluxes not presently covered by MIL-F-14256, with an end goal of accumulating comparative data upon which to base the selection criteria of a Military Procurement Specification.

2. ABSTRACT

Twenty-one manufacturers of fluxes were contacted and twelve found who make water soluble residue fluxes. From these nineteen samples of materials have been obtained. Preliminary testing shows wide variations in soldering efficiencies.

3. PUBLICATIONS, LECTURES, REPORTS, CONFERENCES

Publications: None

Lectures: None

Reports: 1st Monthly Report, June 30, 1965, O.D. Black

2nd Monthly Report, July 30, 1965, O.D. Black

Conferences: 1st Monthly Review; Fort Monmouth, N.J. - June 22, 1965

A. Orlowski, S. Rosen, C.H. Kreck, O.D. Black

2nd Monthly Review; Camden, N.J. - August 4, 1965

A. Orlowski, S. Rosen, C.H. Kreck, O.D. Black

4. FACTUAL DATA

Introduction

The study of water soluble residue fluxes has been started and is proceeding according to the schedule submitted in the Proposal for Standardization Engineering Practices Study of Soldering Flux prepared for:

U.S.A.E.C.
Fort Monmouth, New Jersey
RFP (AMC (E) 28-043-65-00303)

by Central Engineering
Defense Electronics Products
Radio Corporation of America
Camden, New Jersey

submitted 11 March, 1965.

Phase A. Survey of Flux Manufacturers

From commercial literature the names of twenty-one manufacturers were selected as representative. These were contacted and asked whether or not they made such fluxes.

Seventeen replied and twelve indicated that they did, indeed, manufacture such fluxes. From these twelve manufacturers sample lots of nineteen different fluxes were obtained for the tests.

Attached is a letter of the type used.

May 26, 1965

Gentlemen:

In an effort to become more familiar with the various solder fluxes produced for the Electronic Industry we are initiating an evaluation study of those fluxes not presently covered by military specifications MIL-F-14256 and QQ-S-571. Of special interest are the water soluble residue fluxes and flux cored solders.

If you manufacture such materials we would greatly appreciate receiving cost information and technical data. Under the latter we would like to know the chemical classification (hydrazine, glutamic acid-hydrochloride, levulinic acid, etc.), solvents and recommendations for flux application, soldering temperatures and post soldering cleaning.

Very truly yours,

Otis D. Black
Materials Standards
Central Engineering
RCA Building 1-6-2
Camden, New Jersey 08102

gls

Phase A has been completed. It is felt that enough manufacturers have been contacted and enough materials obtained to be representative of all materials of this type.

Phase B. Analysis of Fluxes

This has been started. Five samples were chosen for the following analytical tests:

1. Total solids
2. Infra-red spectrophotometric curve on solids
3. Total acidity and total chlorides
4. Specific gravity
5. Refractive index
6. Boiling curve; distillation curve
7. Presence of heavy metals

These analyses are being conducted in the Analytical Laboratories of Central Engineering.

1. Total solids. This test is being run to determine the percentage of active ingredients in the flux. This will enable us to compare fluxes at the same concentration and it will also furnish data that can be used in formulating controls for purchasing specifications, if so desired. Total solids are found by evaporating 100 grams of the liquid flux to constant weight at 110°C. The temperature might have to be raised if it is found by the boiling curve determination that higher boiling polyols are used in the solvent formulation.

2. Infra-red Spectrophotometric Curve on Solids.

Thoroughly dried flux solids, as obtained from the total solids test, will be ground with potassium bromide to form an intimate, fine powder. This will be pressed into a pellet of proper size and a spectrophotometric curve in the infra-red determined by means of a [redacted] Infracord Spectrophotometer Model.

This test will relate to functional groupings in the flux compounds. While not ordinarily quantitative it will serve to classify the various flux solids qualitatively and will be correlated with data furnished by the manufacturer as well as that obtained from other sources. This will assist in categorizing the various fluxes.

3. Total Acidity and Total Chlorides.

Standard chemical titrations will be run to determine total acidity. The total chlorides and other halides will be run to determine whether or not all of the acid present is hydrochloric. This information will assist in testing for complete flux removal.

4. Specific Gravity.

This test is being run for comparative purposes. It is dependent on the types and amounts of active ingredients and types and amounts of solvents. It will furnish data that can be used to assure the same composition in each different sample of what is purportedly the same flux. This test will be run by means of a simple hydrometer immersed in the sample at a fixed temperature (25°C, 77°F).

5. Refractive Index.

This test is being run for comparative purposes. Similar to the specific gravity test it depends on the types and amounts of active ingredients and types and amounts of solvents. It will furnish data that can assure compositional control. It will be run on each sample at 25°C (77°F) on a Bausch and Lomb Refractometer, Model VD 686.

6. Boiling Curve.

A boiling curve, or distillation range curve, will be run on each sample to indicate the types of solvents blended to make the carrier. This will identify the presence of higher boiling solvents and indicate, at least to some degree, the thermal stability of the flux. Data obtained in this way will be particularly valuable in determining changes in solvent composition from batch to batch.

7. Presence of Heavy Metals.

Standard qualitative analytical tests will be run to determine presence or absence of copper, lead, cadmium, cobalt, nickel, iron, chromium, aluminum, zinc and manganese. This is to make sure that there are no metal cations present which could cause precipitation of difficulty soluble salts which might cause corrosion similar to that produced by zinc oxychloride when zinc chloride fluxes are used.

Phase C. Determination of Solderability

Preliminary edge dip tests for solderability have been run on all nineteen samples and some ideas obtained on relative soldering efficiencies. There is a wide variation in soldering efficiencies among the various fluxes heavily oxidized boards.

The equipment required for the Electronics Command Meniscus Soldering Test as developed by G.E. has kindly been loaned to us by the General Electric Laboratory at Schenectady. This has been set up, sample boards prepared and initial tests begun.

5. CONCLUSIONS

Phase A has been completed. There are enough manufacturers of this type of flux and enough different brands to insure that this will continue to be a commercially available item. The manufacturers are large enough and reputable enough to insure that specification for composition would be consistently met.

There are considerable differences in soldering efficiencies among the fluxes.

6. PROGRAM FOR NEXT INTERVAL

The next three months will be spent in finishing the analytical work, Phase B, determining solderability, Phase C, preparing samples, Phase D and starting the soldering of samples for the cleaning tests, Phase E.

Phase B has been discussed above and will be finished in this time interval.

Phase C. Determination of Solderability

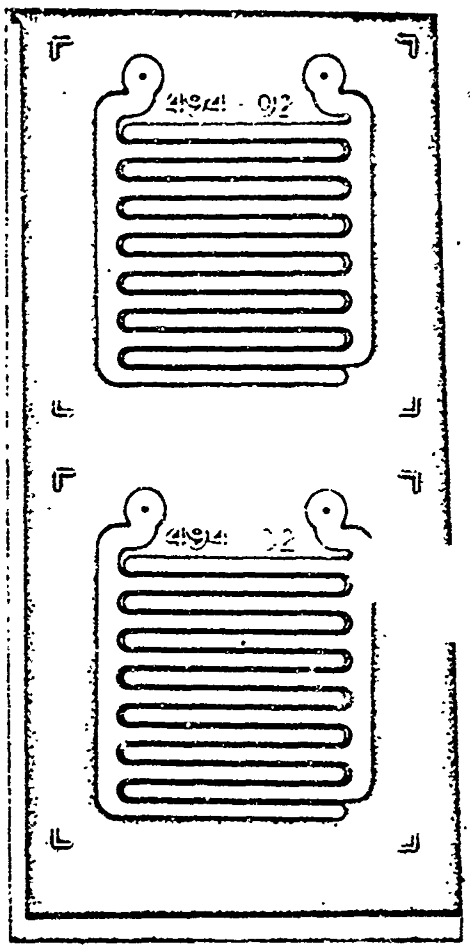
The relative solderabilities of each of the five selected fluxes will be determined by means of the IPC Edge Dip Method and by means of the Electronics Command Meniscus Test for Solderability as developed by the General Electric Co. These tests will also be finished in this period.

Phase D. Preparation of Samples

Samples to be fluxed and soldered will be etched copper test patterns of the RCA type Pattern 494-02, a picture of which is attached. This is a comb-type pattern with an effective path width of .032"; the pattern is 13" long. Any decrease in insulation resistance is readily measurable and any corrosion products are readily visible. This particular pattern was chosen over some that are in Military Specifications, particularly the one in MIL-P-55110 because test data have shown that it is more critical and because it takes up less space.

RCA has extensive data on test samples made with this pattern in which many types of fluxes were tried. Direct correlation of results can be made with those obtained when alcohol rosin fluxes approved under MIL-F-14256 were used. While RCA recommends the use of this pattern, a different pattern will be used if required by the US Army Electronics Command.

The copper surfaces will be prepared for soldering by a standard cleaning procedure in order to produce uniform results. RCA has had excellent results with the following method:


4194 - 02

Scrub with an abrasive cleanser until the copper shows no water break.

Rinse thoroughly with running tap water.

30 second immersion in dilute cupric/hydrochloric acid.

60 second rinse with running tap water.

30 second immersion in 1-3 hydrochloric acid.

60 second rinse with running tap water.

Thorough rinse in deionized water.

Blow dry with oil free, filtered compressed air.

Other methods of preparing boards to give good initial solderability will be evaluated, including that specified by the General Electric Company and described in Contract DA-36-039-AMC-00008(E). If no better method is found the RCA technique will be used.

At least 36 sample boards of single sided copper clad glass epoxy will be prepared for each of the five fluxes chosen, or 180 in all. This will give 6 test boards for each of the six types of cleaning, the types of cleaning being described in Phase F.

Materials will be added to each of the fluxes to assist in determining the effectiveness of flux residue removal. At least three fluorescent dyes and two chemical compounds, extremely low concentrations of which fluoresce under ultra violet light, will be evaluated. While more sophisticated techniques such as radioactive tracers may yield more precise results, it is felt that there is a definite need to develop and standardize a residue-determining technique which will be sufficiently accurate for practical applications by small business concerns.

Boards will be fluxed by brushing and then dried in accordance with the manufacturers recommendations. This technique will be modified if it is found that spattering results from flux application or subsequent soldering operation.

This will be finished in this period.

Phase E. Soldering

The soldering operation will be performed by manually contacting the samples onto the surface of a 60/40 composition solder bath maintained at a temperature of 500°F. A 10 second dwell time will be used. In each case the solder surface will be cleaned before the sample is dipped. RCA has found that this combination of time and temperature will produce excellent soldering results. However, this procedure will be modified if flux vendors' data indicate changes in time or temperature. Tests will also be conducted at 460° and 525° to determine the temperature of most effective flux action.

This will be started during this period.

Phase F. Cleaning

The following cleaning cycles will be evaluated:

1. 5 minutes rinse in running tap water, followed by a rinse in deionized water and drying.
2. 15 minutes rinse in running tap water, followed by a rinse in deionized water and drying.

3. 2 minute soak in detergent solution, followed by 5 min. rinse in running tap water, followed by a rinse in deionized water and drying.
4. Same as #1 with light brushing.
5. Same as #2 with light brushing.
6. Same as #3 with light brushing.

If, upon testing, it is found that these cleaning methods do not completely remove the flux residue, the cleaning will be tried again, using moderately warm rinse water (120°-140°F). For good production, however, the flux residue should be readily removable by running water at approximately room temperature.

This will be started during this period.

7. KEY PERSONNEL

F. X. Thomson — Approx. 20 hours

Leader, Material Analysis and Metallic Materials

Education: BS in Chemistry, 1936, Providence College; MS in Chemistry, 1938, Rhode Island State University. He took additional graduate work in organic chemistry at Columbia and Princeton Universities from 1939 to 1942.

Mr. Thomson is responsible for the operation of the mechanical-metallurgical laboratories and for supplying RCA with control documentation and consultation in the fields of welding, casting, forming, heat treating, etc. His activity is staffed and facilitated to carry out extensive investigations into the structural properties and behavior of metal, and to perform analytical and physical research in metallurgical areas.

From 1938 to 1942, Mr. Thomson was an Associate Professor of Chemistry at Seton Hall University. From 1942 to 1945, Mr. Thomson was employed by Hoffman LaRoche, Inc. and Merck & Co. He was engaged in analytical and physical research and development on vitamins, pharmaceuticals and drugs. Mr. Thomson joined RCA in 1945 where he was employed as an engineer in the Chemical and Physical Laboratories.

Mr. Thomson is a member of the American Chemical Society and the Society for Applied Spectroscopy. He is the author of several technical papers.

O. D. Black (Dr.) Approx. 92 hours

Research and Development Engineer, Materials Standards

Dr. Black received the BA degree in 1938, the M.Sc. in 1939, and the Ph.D. in 1942, all in Physical Chemistry, from Ohio State University. He joined RCA in 1942 in the Chemical and Physical Laboratories in Camden as a Research and Development Engineer. He has been engaged in a number of studies involving plastics, ceramics, rubber and allied fields and mycology. Since 1947 he has been primarily engaged in research and development of materials and processes involved in the manufacture of printed circuits. Dr. Black is now a member of the Materials Standards section of Central Engineering, with general responsibility for materials such as laminates, etchants and cleaners and for the processes such as cleaning, silk screening, etching and soldering in a factory with a capacity of approximately 10,000 boards per day. He coordinates the activities of specialists as required and has complete responsibility for testing and approval of copper clad laminates used by the Defense Electronic Products Division of RCA. Dr. Black has contributed to various technical publications and has a number of patents in the printed wiring field. He is a member of Sigma Xi, Phi Beta Kappa and Phi Lambda Upsilon and is listed in American Men of Science. His current military clearance is "Secret"